PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

(Chapter II of the Patent Cooperation Treaty)

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference F17736 GSK	**FOR FURTHER ACTION**	See Form PCT/IPEA/416
International application No. PCT/IB2004/000080	International filing date *(day/month/year)* 15.01.2004	Priority date *(day/month/year)* 17.01.2003
International Patent Classification (IPC) or national classification and IPC B01J31/40		
Applicant SASOL TECHNOLOGY (PROPRIETARY) LIMITED et al.		

1. This report is the international preliminary examination report, established by this International Preliminary Examining Authority under Article 35 and transmitted to the applicant according to Article 36.
2. This REPORT consists of a total of 5 sheets, including this cover sheet.
3. This report is also accompanied by ANNEXES, comprising:
   a. ☒ *sent to the applicant and to the International Bureau)* a total of 5 sheets, as follows:
      ☐ sheets of the description, claims and/or drawings which have been amended and are the basis of this report and/or sheets containing rectifications authorized by this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions).
      ☐ sheets which supersede earlier sheets, but which this Authority considers contain an amendment that goes beyond the disclosure in the international application as filed, as indicated in item 4 of Box No. I and the Supplemental Box.
   b. ☐ *(sent to the International Bureau only)* a total of (indicate type and number of electronic carrier(s)) , containing a sequence listing and/or tables related thereto, in computer readable form only, as indicated in the Supplemental Box Relating to Sequence Listing (see Section 802 of the Administrative Instructions).

4. This report contains indications relating to the following items:

☒	Box No. I	Basis of the opinion
☐	Box No. II	Priority
☐	Box No. III	Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
☐	Box No. IV	Lack of unity of invention
☒	Box No. V	Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
☐	Box No. VI	Certain documents cited
☐	Box No. VII	Certain defects in the international application
☐	Box No. VIII	Certain observations on the international application

Date of submission of the demand	Date of completion of this report
16.11.2004	27.01.2005
Name and mailing address of the international preliminary examining authority: European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Authorized Officer Klaes, D Telephone No. +49 89 2399-7335

INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

International application No.
PCT/IB2004/000080

Box No. I Basis of the report

1. With regard to the **language**, this report is based on the international application in the language in which it was filed, unless otherwise indicated under this item.

   ☐ This report is based on translations from the original language into the following language , which is the language of a translation furnished for the purposes of:
   - ☐ international search (under Rules 12.3 and 23.1(b))
   - ☐ publication of the international application (under Rule 12.4)
   - ☐ international preliminary examination (under Rules 55.2 and/or 55.3)

2. With regard to the **elements*** of the international application, this report is based on *(replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report)*:

Description, Pages	
1-14	as originally filed
Claims, Numbers	
1-28	received on 18.11.2004 with letter of 16.11.2004
Drawings, Sheets	
1/3-3/3	as originally filed

   ☐ a sequence listing and/or any related table(s) - see Supplemental Box Relating to Sequence Listing

3. ☐ The amendments have resulted in the cancellation of:
   - ☐ the description, pages
   - ☐ the claims, Nos.
   - ☐ the drawings, sheets/figs
   - ☐ the sequence listing *(specify)*:
   - ☐ any table(s) related to sequence listing *(specify)*:

4. ☐ This report has been established as if (some of) the amendments annexed to this report and listed below had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).
   - ☐ the description, pages
   - ☐ the claims, Nos.
   - ☐ the drawings, sheets/figs
   - ☐ the sequence listing *(specify)*:
   - ☐ any table(s) related to sequence listing *(specify)*:

* *If item 4 applies, some or all of these sheets may be marked "superseded."*

Box No. V Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes:	Claims	1-28
	No:	Claims	
Inventive step (IS)	Yes:	Claims	1-28
	No:	Claims	
Industrial applicability (IA)	Yes:	Claims	1-28
	No:	Claims	

2. Citations and explanations (Rule 70.7):

see separate sheet

Re Item V

Reasoned statement with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

In this communication the documents cited in the search report are referred to. The numbering is based on their order of appearance therein.

D1 claims a method for recovering a group VIII metal complex which has been used in hydroformylation reactions. Examples 7 discloses a hydroformylation reaction which is catalyzed by $Rh(acac)(CO)_2$ in presence of $(n\text{-}C_{18}H_{37})_3P$. To recover the catalyst methanol is added to the reaction mixture. The mixture is cooled to a temperature of from 4 to 5°C, whereby a rhodium complex precipitates. The precipitate is subjected to filtration. The same process to recover a Co-CO-phosphine complex is also known (example 4). Instead of methanol ethanol, propanol and n-butyl alcohol can be used (p. 7, l. 9). The reaction in general is carried out within a range from 20-50°C (p. 7, l. 14-15). The precipitated complex can be separated by filtration, centrifugal filtration or centrifugal separation (p. 7, l. 16-18).

D2 discloses the recovery of a catalyst comprising group VIII noble metal and a phosphite ligand by mixing the catalyst liquid (which has been obtained by distillation of a reaction mixture after hydroformylation reaction) with an organic solvent and recovering the catalyst as crystals by a filtration method. As organic solvent alcohols of the carbon number 1-8 like methanol, ethanol and ethylene glycol are either used alone or as mixture. The crystallisation is carried out at a temperature in a range from 0°-70°C.

D3 discloses the recovery of Rhodium-phosphine complexes which have been used in hydroformylation reactions. Example 1 describes the process in detail. The reaction mixture is concentrated by distillation and ethanol and an aqueous solution of NaOH are added at 70°C. The mixture is stirred at this temperature in an atmosphere of air and the rhodium complex precipitates. The complex can be separated by usual methods like filtration, centrifugal filtration and centrifugal separation (column 6, l. 47-52).

The presence of aldols, acetals and esters is disclosed implicitly in D1-D3 as they are known byproducts of hydroformylation reactions.

By limiting the scope of the independent claims 1 and 14 to bicyclic tertiary phosphine ligands novelty has been installed for claims 1-28 (Art. 33 (2) PCT).

As the solubility of the catalyst depends on the ligands it is not obvious that the teaching of D1-D3 (D1: phosphines bearing "simple" alkyl substituents; D2: phosphites; D3: triarylphosphines (preferred embodiment)) can be extended to bicyclic tertiary phosphines. D1 even teaches away from the invention. According to D1 the sum of the carbon number of the three alkyl substituent must be at least 42. For ligands of formula (I) and (II) (claims 2, present application) good results can be achieved although the sum is only 28.

Therefore, inventive step can be acknowledged for claims 1-28 (Art. 33 (3) PCT).

Claims 1-28 are industrially applicable (Art. 33 (4) PCT).

Re Item VII

Certain defects in the international application

1.1 Contrary to the requirements of Rule 5.1(a)(ii) PCT, the relevant background art disclosed in the documents D1-D3 is not mentioned in the description, nor are these documents identified therein. The application does not disclose a single prior art document.

1.2 The description is not in line with the claims on file.

PCT/IB 2004/80

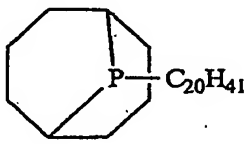
CLAIMS

1. A process for recovering an active catalyst component from a process stream, which process includes

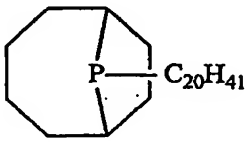
admixing an alcohol component comprising at least one C_1 to C_{10} alcohol, with a process stream containing a dissolved active catalyst component comprising a metal/carbon monoxide/ligand complex and free ligand, and wherein the ligand is a bicyclic tertiary phosphine, to form solid active catalyst component comprising metal/carbon monoxide/ligand complex and free ligand; and

recovering the solid active catalyst component from a residual alcohol-rich phase which contains also inactive phosphine ligand oxides.

2. A process according to Claim 1, wherein the process stream is a hydroformylation process heavies purge stream, and wherein, in respect of the active catalyst component, the metal is cobalt and the ligand is a mixture of a 9-phosphabicyclo[3.3.1]nonane and a 9-phosphabicyclo[4.2.1]nonane represented by formulae (I) and (II) respectively:



I



II

3. A process according to Claim 1 or Claim 2, wherein the alcohol component comprises at least one C_1 to C_{10} monohydric alcohol and/or at least one C_1 to C_{10} dihydric alcohol.

4. A process according to Claim 3, wherein the alcohol of the alcohol component is in concentrated or undiluted form.

5. A process according to Claim 4, wherein the alcohol component comprises a concentrated monohydric alcohol having 1 to 3 carbon atoms.

6. A process according to Claim 5, wherein the monohydric alcohol of the alcohol component is concentrated methanol or concentrated ethanol.

7. A process according to Claim 4, wherein the alcohol component comprises a concentrated dihydric alcohol having 2 to 8 carbon atoms.

8. A process according to Claim 7, wherein the dihydric alcohol component of the alcohol component is concentrated ethylene glycol or concentrated propylene glycol.

9. A process according to Claim 4, wherein the alcohol component comprises a mixture of at least two concentrated C_1 to C_{10} alcohols.

10. A process according to any one of Claims 1 to 9 inclusive, wherein the alcohol component, on admixture thereof with the process stream, is at a temperature below room temperature and above its freezing temperature.

11. A process according to Claim 10, wherein the alcohol component is at a temperature below 0°C.

12. A process according to any one of Claims 1 to 11 inclusive, wherein the formation of the solid active catalyst component is by means of precipitation or crystallization.

13. A process according to any one of Claims 1 to 12 inclusive, which includes subjecting the process stream, after admixture of the alcohol component therewith, to centrifugation.

14. A hydroformylation process, which includes

reacting, in a reaction zone and in the presence of a hydroformylation catalyst comprising a metal/carbon monoxide/ligand complex in which the ligand is a bicyclic tertiary phosphine, and at elevated temperature and pressure, an olefin-containing feedstock with carbon monoxide and hydrogen, to form aldehydes and/or alcohols;

withdrawing a reaction mixture comprising the alcohols, the aldehydes, unreacted feedstock, a catalyst residue which includes (i) an active catalyst component comprising metal/carbon monoxide/ligand complex and free ligand, and (ii) inactive phosphine ligand oxides, heavies and, optionally, unreacted gaseous reactants, from the reaction zone;

in a separation zone, separating a gaseous phase from a liquid phase comprising the aldehydes, alcohols, unreacted feedstock, the heavies and the catalyst residue;

in a distillation zone, subjecting the liquid phase to distillation;

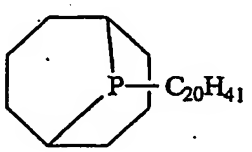
withdrawing from the distillation zone, as an overheads component, the alcohols, aldehydes and unreacted feedstock;

withdrawing from the distillation zone, as a bottoms component, the heavies and the catalyst residue, at least some of which is in solution;

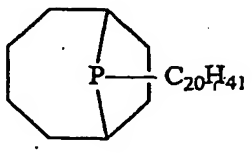
admixing an alcohol component comprising at least one C_1 to C_{10} alcohol, with at least a portion of the bottoms component to form solid active catalyst component comprising metal/carbon monoxide/ligand complex and free ligand; and

recovering the solid active catalyst component from a residual alcohol-rich phase which contains also the inactive phosphine ligand oxides.

15. A process according to Claim 14, wherein, in respect of the hydroformylation catalyst, the metal is cobalt, and the ligand is a mixture of a 9-phosphabicyclo[3.3.1]nonane and a 9-phosphabicyclo[4.2.1]nonane represented by formulae (I) and (II) respectively;



I



II

16. A process according to Claim 14 or Claim 15, which includes recycling a portion of the bottoms component to the reaction zone, with the portion thereof that is admixed with the alcohol component thus constituting a heavies purge stream that is withdrawn.

17. A process according to Claim 16, wherein no dilution of the bottoms component, at least prior to the withdrawal of the heavies purge stream therefrom, or of the heavies purge stream, with a saturated or unsaturated aliphatic hydrocarbon having 3 to 20 carbon atoms or with an aromatic or hydrocarbyl-substituted aromatic hydrocarbon having from 6 to 22 carbon atoms, takes place.

18. A process according to any one of Claims 14 to 17 inclusive, wherein the alcohol component comprises at least one C_1 to C_{10} monohydric alcohol and/or at least one C_1 to C_{10} dihydric alcohol.

19. A process according to Claim 18, wherein the alcohol of the alcohol component is in concentrated or undiluted form.

20. A process according to Claim 19, wherein the alcohol component comprises a concentrated monohydric alcohol having 1 to 3 carbon atoms.

21. A process according to Claim 20, wherein the monohydric alcohol of the alcohol component is concentrated methanol or concentrated ethanol.

22. A process according to Claim 19, wherein the alcohol component comprises a concentrated dihydric alcohol having 2 to 8 carbon atoms.

23. A process according to Claim 22, wherein the dihydric alcohol component of the alcohol component is concentrated ethylene glycol or concentrated propylene glycol.

24. A process accoding to Claim 19, wherein the alcohol component comprises a mixture of at least two concentrated C_1 to C_{10} alcohols.

25. A process according to any one of Claims 14 to 24 inclusive, wherein the alcohol component, on admixture thereof with the bottoms component, is at a temperature below room temperature and above its freezing temperature.

26. A process according to Claim 25, wherein the alcohol component is at a temperature below 0°C.

27. A process according to any one of Claims 14 to 26 inclusive, wherein the formation of the solid active catalyst component is by means of precipitation or crystallization.

28. A process according to any one of Claims 14 to 27 inclusive, which includes subjecting the bottoms component, after admixture of the alcohol component therewith, to centrifugation.